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Responses of an Electron Capture Detector to Some Inert, Non-toxic Low Molecular Weight Compounds

by

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Naval Weapons Center
CHINA LAKE, CALIFORNIA ■ DECEMBER 1971

ABSTRACT

Materials capable of scavenging ambient electrons have found application in gaseous dielectrics, in ionospheric electron depletion, and as inhibitors of some of the chemical reactions following radiolysis. Responses of a tritium-based electron-capture detector are measured in order to determine what compounds are effective scavengers, particularly those that are inert, non-toxic and of low molecular weight. A detailed description is given of the apparatus. Vapor samples are expanded successively between two interconnecting chambers to achieve a sufficient dilution for a measurable, linear response, and the apparatus has the capability of handling many chemically reactive compounds, if necessary. A new material, comparable in effect to sulfur hexafluoride is perchloryl fluoride, although it would seem that further chemical searches will not improve greatly on SF_6.

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FOREWORD

This study was conducted at the U. S. Naval Weapons Center under funds from Naval Material Command under Project No. ZR01101, Task Area No. ZR0110101 from December 1970 to January 1971. It is part of a continuing program concerned with the physical and chemical properties of electronegative materials and follows on two earlier reports: (1) "Electron Acceptors as Electron Scavengers in the Ionosphere," U. S. Naval Ordnance Test Station, China Lake, California; NAVWEPS Report 8764, NOTS TP 3846, July 1965 (CONFIDENTIAL); and (2) "Concerning the Electron Affinities of Sulfur Hexafluoride and some Other Electron Scavenging Molecules," J. CHEM. PHYS., Vol. 55 (1971), pp. 3468-3471.

The report was reviewed for technical accuracy by D. W. Moore and W. P. Norris.

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INTRODUCTION

Materials capable of scavenging ambient electrons have found application in gaseous dielectrics, in ionospheric electron depletion (Ref. 1), and as inhibitors of some of the chemical reactions following radiolysis (Ref. 2). They have been the basis of proposals such as a means of alleviating the energy of tornadoes (Ref. 3), in jamming over-the-horizon radar communications (Ref. 4), and in suppressing reentry radar wakes (Ref. 5).

A detailed study of the scavenging process would examine the influence of temperature, pressure, third-body effects on the dissociative and non-dissociative capture cross-sections. This was not our aim. Rather, we wanted to know what materials are effective electron scavengers, particularly those that are inert, nontoxic and of low molecular weight. For this purpose measurements were made of the response of a tritium-based electron-capture-detector operating at atmospheric pressure in nitrogen. The results are expressed in terms of the response ratio compared with that produced by the same molar quantity of sulfur hexafluoride, and they supplement the useful measurements of Clemons and Altshuller (Ref. 6). The technique employed by these authors for very active scavengers was to sample the material from a large, single, diluted volume (250 liters). For the present measurements a glass-Teflon dilution apparatus was constructed. Two interconnecting chambers, which could be evacuated or filled with carrier gas, permitted successive transfer of material from one chamber at atmospheric pressure to the other under vacuum, and samples were examined during the dilution steps. Although numerous appropriate compounds were examined, the measurements are not exhaustive and the apparatus is constructed to handle chemically reactive materials if necessary.

EXPERIMENTAL

A Wilkens Aerograph Model 600C chromatograph and associated electrometer was used. The signal and the standing current was measured by a 0-1 MV, 1 second response Hewlett Packard recorder. At carrier gas flow rates of 40 ml per minute standing currents of 3.95 to 3.70 MV were recorded. Normal daily variations of ±0.03 MV were found. Column temperatures were 60°, 38° and 25° with corresponding injector temperatures of 125°, 38° and 25°C. Peak areas were measured by a Wilkens

Aerograph Model 470 Integrator which has a linear counting rate of 60,000 counts per minute to a maximum signal of 50 MV. The tritium foil was sonically cleaned with 5% methanolic potassium hydroxide for thirty minutes, rinsed with ethanol, water, and dried. The foil was not removed from the electrode although the Teflon plug was removed for ready access to the foil cavity.

CARRIER GAS

A nitrogen-hydrogen gas mixture (97% N_2 and 3% H_2) from the Air Reduction Company was used as carrier gas. Purification was necessary to remove water and oxygen and to obtain a standing current that remained constant for several days. Positive hydrocarbon peaks were eliminated when an activated charcoal trap was installed. A copper furnace heated to 280°C was used to remove traces of oxygen. The water in the carrier gas and that formed by the reaction of copper oxide and hydrogen was removed by a 5A molecular sieve trap.

Carrier gas flow regulation was accomplished by restrictor tubing (0.010 inch ID, 1/16 inch OD stainless steel tubing) and a Moore Products Company constant differential type flow controller Model 8/M. Gas used to pressurize the dilution flask passed through three sections of restrictor tubing which totaled eight inches in length. After passing through a one inch section the gas stream was divided, one section going to a five inch length of restrictor tubing, the other leading to the control side of the differential flow regulator via eight feet of the restrictor tubing and continuing through the gas sample valve into the gas chromatographic column (see Fig. 1).

The gas flowing through the five inch restrictor was again divided, part going to another short restrictor and the gas dilution flask, while part passed through eighteen feet of the 0.010 inch ID tubing before being used as purge gas for the column and gas sampling valve.

The pressure of the gas entering the gas sampling valve was 20 psig when the ten foot squalane on Teflon 6 column was in the system and 9.7 psig when the 24 inch long restrictor was installed in place of the column. Carrier gas flow rates at the detector was 40 ml per minute.

GAS CHROMATOGRAPHIC COLUMN

A 1/8-inch OD Teflon column 10.9 feet in length containing 9.98 grams of column packing could be used to separate solvents and impurities from the compounds of interest. In those cases where strongly oxidizing materials were examined the column was removed and the 24 inch flow restrictor used in its place. The flow restrictor was required to avoid excessive flow changes during the operation of the gas sampling valve.

Squalane which had been hydrogenated for 60 hours at 50 psig with platinum oxide catalyst was used as stationary phase. Teflon 6 was

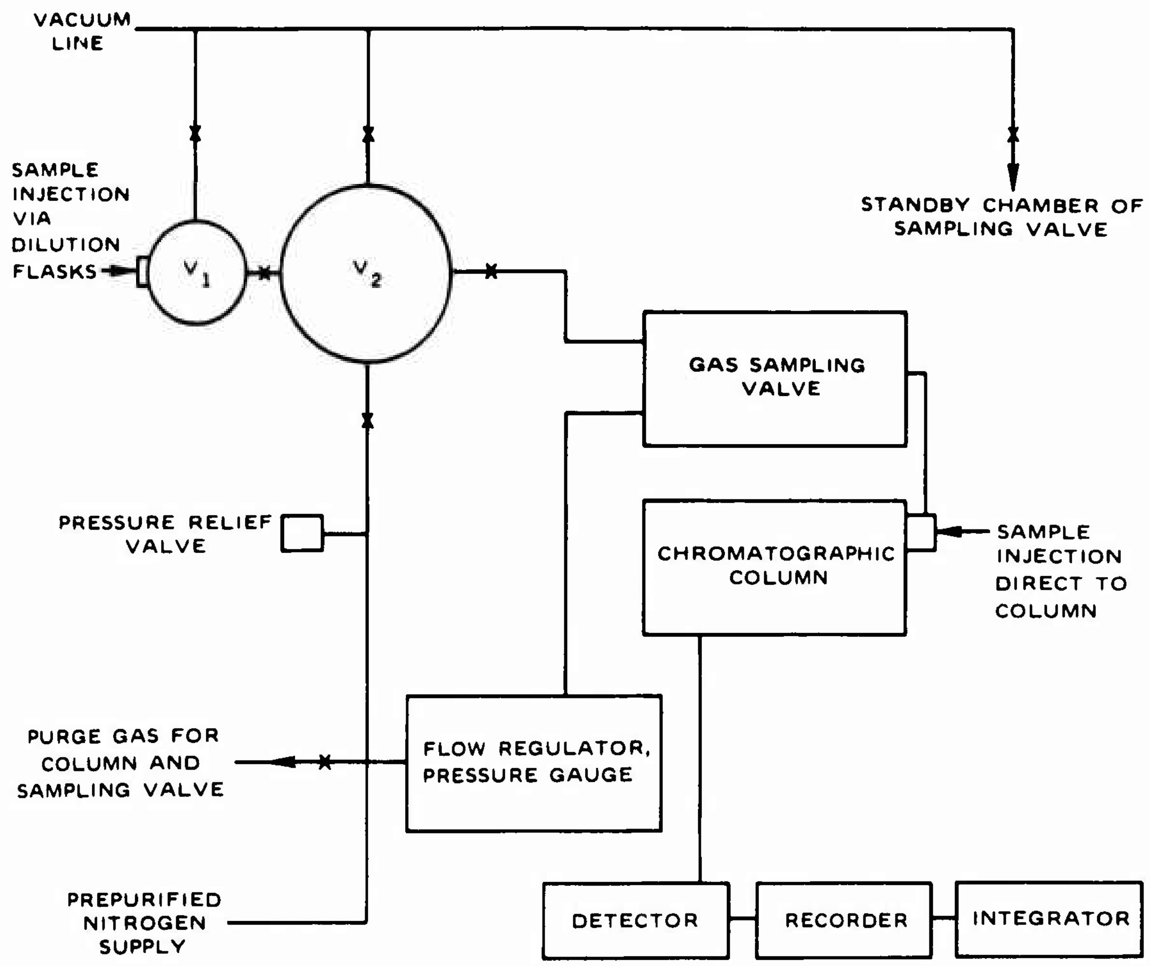

FIG. 1. Schematic Diagram of Measuring Apparatus.

coated with 2% hydrogenated squalane after the method described by Kirkland (Ref. 7). Filling of the Teflon tubing was accomplished by vibrating the feed tray and tubing continuously as the cooled column packing was poured on the tray. This gentle vibration was used to avoid closing the pores of the Teflon 6, a process suspected to occur in sieving operations (Ref. 8).

The Teflon column was employed so as to avoid chemical losses of fluorine containing substances (Ref. 9). It was encased in 1/4-inch OD copper tubing and purged with carrier gas since it was found to be porous to oxygen, altering the baseline and detector response. [The porosity of Teflon has been used to prepare standard concentrations of other gases in a flowing stream (Ref. 10)]. The same gas stream purged the Carle Micro Volume gas sampling valve which had sample loops of 1.0 and 0.1 ml capacity. The purge gas flow rate was 8-9 ml per minute.

SAMPLE PREPARATION

Gas samples were taken by means of a syringe from the Hamilton Lecture Bottle Septum, part number 840, as a slow stream of the gas flowed through a narrow two foot vent line. For liquid samples ten microliters were diluted to 50 ml with cyclohexane, a solvent with negligible response. After thorough mixing ten microliters of this were diluted to 50 ml with cyclohexane to obtain solutions in the 10^{-12} molar concentration range.

SAMPLE INTRODUCTION

Compounds with low response were injected directly as gas, liquid or solution into the gas chromatograph inlet while those of higher response were introduced via the dilution flask. Some volatile liquids were sampled directly in small quantities. A limitation for some liquid compounds of high response was that trace quantities became adsorbed onto the glass walls of the apparatus and could be detected even after several nitrogen flushings.

Reproducible introduction of small fixed quantities of liquid samples was achieved only through the use of a solvent flush technique (Ref. 11). Difficulties arose from the compression of the gas found between the syringe plunger and the liquid sample. The technique developed to minimize this effect required pure solvent be drawn into the syringe, followed by a small quantity of air, the liquid sample whose volume was carefully measured, and some additional air. The residual solvent in the needle after injection had to be less than that originally taken.

GAS DILUTION

The gas dilution apparatus was an all Teflon-glass system with two interconnecting volumes of 4.03cc (V_1) and 526.3cc (V_2). Lab Crest quick opening valves were used throughout the system. Each volume could be evacuated independently and V_2 could be pressurized with carrier gas to atmospheric pressure. Mixing of the gases in V_2 was accomplished with a large Teflon coated stirring bar.

Operating procedure was as follows: (1) V_1 was filled to atmospheric pressure via V_2 before introduction of the sample into V_1 through a Teflon coated septum (available from Canton Bio-Medical Products, P. O. Box 154, Swarthmore, PA, 19081). (2) V_2 was evacuated to 0.025 torr, the sample expanded from V_1 into V_2, and V_2 brought to atmospheric pressure with carrier gas. (3) Evacuation of either loop of the Micro Volume gas sampling valve and opening of a tap between V_2 and the loop permitted introduction of either 0.1 ml or 1.0 ml of the sample vapor to the chromatograph. (4) Further dilutions were possible by evacuation of V_1, expansion of the sample into that volume, evacuation of V_2, expansion from V_1 into V_2, and return to atmospheric pressure for sampling. This could be repeated as many times as required to obtain a gas chromatographic elution peak whose height was within the linear range of the detector [approximately 1 MV or 30% of the standing current (Ref. 12)]. Smaller peak heights were generally used as it has been reported that there is a ten percent error when the standing current is reduced by 10%; approximately 0.4 MV (Ref. 13).

The apparatus was evacuated and refilled several times to obtain low zero readings after each sample. Repeated filling and evacuation of the smaller volume alone would have been desirable but was not possible in the present equipment. Zero readings obtained prior to final evacuation and transfer seldom exceeded 0.1 of that anticipated for the sample. The data recorded is the average taken for several runs with each component at two or three different concentrations.

RESULTS AND DISCUSSION

The limit of detection for sulfur hexafluoride was 10^{-15} g mole, that is for a response approximately five times that obtained from a typical cyclohexane solvent blank. Near the limit of detection (within an order of magnitude) for this and for other compounds, the response was linearly related to concentration. The results of Table 1 are expressed as comparisons with sulfur hexafluoride for the same molar quantities, within this concentration region. At elevated temperatures a diminished response was obtained from selenium hexafluoride using the squalane column, and from trifluoramine and trifluoramine oxide using the replacement 24-inch flow restrictor, a probable result of chemical reaction. In the neighborhood of room temperature the first

TABLE 1. Response of Some Electronegative Compounds to an Electron Capture Detector.

Compound	Form of sample injection	Column	Column temperature	Response (compared with SF_6 on a molar basis)
Freon 11 (CCl_3F)	Vapor	Squalane	25	0.6
Carbon tetrachloride	Cyclohexane solution	Squalane	60	0.4
Sulfuryl fluoride	Vapor	Squalane	25	0.3×10^{-2}
Perchloryl fluoride	Vapor	Squalane	25	0.4
Oxygen	Vapor	Squalane	25	0.5×10^{-5}
Sulfur dioxide	Vapor	Squalane	25	0.8×10^{-3}
Carbon disulfide	Cyclohexane solution	Squalane	60	0.2×10^{-3}
1,2-Dichloroperfluoro-cyclopentene	Cyclohexane solution	Squalane	60	0.2
Chloropentafluorobenzene	Cyclohexane solution	Squalane	60	0.1
Hexafluorobenzene	Cyclohexane solution	Squalane	60	0.7×10^{-1}
Perfluorotoluene	Cyclohexane solution	Squalane	60	0.3×10^{-1}
Selenium hexafluoride	Cyclohexane solution	Squalane	60	0.4×10^{-6}
Selenium hexafluoride	Cyclohexane solution	Squalane	25	0.7×10^{-2}
Tellurium hexafluoride	Cyclohexane solution	Squalane	25	0.4×10^{-2}
Tungsten hexafluoride	Cyclohexane solution	Squalane	60	0.7×10^{-7}
Phosphoryl chloride	Cyclohexane solution	Squalane	60	0.8×10^{-3}
Trifluoramine oxide	Vapor	Flow restrictor	60	0.2×10^{-6}
Trifluoramine oxide	Vapor	Flow restrictor	25	0.7×10^{-3}
Trifluoramine	Vapor	Flow restrictor	60	1.0×10^{-7}
Trifluoramine	Vapor	Flow restrictor	25	1.0×10^{-4}
Nitrosyl chloride	Vapor	Squalane	25	1.0×10^{-5}
Silicon tetrachloride	Cyclohexane solution	Squalane	60	1.0×10^{-6}

two showed no change on warming and were assumed to be true responses. Perchloryl fluoride, when left in contact with the steel loop of the gas sampling valve for more than ten minutes gave erratic results whereas on rapid handling it showed repeatable responses that were proportional to concentration. Reaction of the minute quantities of compound with the steel or perhaps traces of impurity in the valve is suspected. Where they overlap the present measurements are in moderate agreement with earlier results, certainly with respect to the order of magnitude, although carbon tetrachloride has been reported to show a 10% greater response than suflur hexafluoride (Ref. 6). Carbon tetrafluoride and nitrous oxide are not included in the Table and produced responses less effective than silicon tetrachloride.

Inferences from these results to conditions other than those studied are of course suspect. Many of the compounds showed very sensitive responses, for example perchloryl fluoride deserves further detailed study. The compounds selected were not exhaustive, for example nitryl fluoride and chloride, which were incompatible with the materials of the apparatus, and difluorodiodomethane were not examined. The main conclusion though is that none of the scavengers were superior to sulfur hexafluoride and it would seem that further searches will not improve on this material greatly. Responses on the electron capture detector larger by an order of magnitude than SF_6 seem unlikely, and certainly not on a weight-for-weight basis, for inert, non-toxic compounds. Information pertinent to the electron affinity of sulfur hexafluoride and other scavenging materials appears elsewhere (Ref. 14).

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Perchloryl Fluoride						

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